Proposed addition to Chapter 5 Quality Systems Appendix D – Water Asbestos Testing

NOTE: This proposal will <u>not</u> be considered for voting or discussion at NELAC VI. It is provided for future discussion at the next NELAC Interim meeting (NELAC VIi).

NELAC Chapter 5 Quality Systems - Appendix D - Essential Quality Control Requirements

D.7 WATER ASBESTOS TESTING

These standards apply to laboratories undertaking the examination of asbestos in drinking water and wastewater samples by the transmission electron microscope (TEM) analysis. These procedures for asbestos analysis involve the sample preparation followed by detection of the chrysotile asbestos.

D.7.1 Negative Controls

a) Blank determinations should be made prior to sample collection. When using polyethylene bottles, one bottle from each batch, or a minimum of one from each 24, should be tested for background level. When using glass bottles, four bottles from each 24 should be tested. An acceptable bottle blank level is defined as <0.01 MFL > 10 µm. (Section 8.2, Method 100.2)

b) A process blank sample consisting of fiber-free water should be run before the first field sample. The quantity of water should be $10 mL for 25 mm diameter filter and $50 mL for a 47 mm diameter filter. (Section 11.8, Method 100.2)

D.7.2 Test Variability/Reproducibility

a) Replicate - Shall be performed at a frequency of 1 per 100 samples to conform with the 1.5Xpoisson std. Dev. (Table 2., Method 100.2)

b) Duplicate - Shall be performed at a frequency of 1 per 100 samples to conform with 2Xpoisson standard deviation (Table 2, Method 100.2)

D.7.3 Other Quality Control Measures

a) Analysis of standards shall be performed at a frequency of 10% for the purpose of training and comparison with unknowns. (Table 2, Method 100.2)

b) Analysis of SRM shall be performed at a frequency of one per analyst per year and conform with 1.5Xpoisson std. Dev. (Table 2, Method 100.2)

D.7.4 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Demonstration of Capability - (section 5.10.2.1) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel or method.

b) Proficiency Test Samples - The results of such analysis (5.4.2.j or 5.5.3.4) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.7.5 Asbestos Measurement System Calibration

Due to the stability and response nature of modern asbestos measurement instrumentation, it is not typically necessary to calibrate these systems in the day of use manner done so for some types of chemical measurement instrumentation. This section will address those practices that are necessary for proper calibration and those requirements of section 5.9.4.2 (Instrument Calibrations) that are not applicable to some types of asbestos measurement instrumentation.

a) Magnification Calibration

Magnification calibration must be done at the fluorescent screen and must be performed at the magnification used for fiber counting, generally 10,000 and 20,000X. A logbook must be maintained with the dates of the calibration recorded. It is recommended that calibrations be performed monthly to establish the stability of the magnification. (Section 10.1, Method 100.2)

b) Camera Constant

The camera length of the TEM in the electron diffraction (ED) mode must be calibrated before ED patterns of unknown samples are observed. The stage must be at the eucentric position for this calibration. The camera constant calculated for that particular instrument can then be used for ED patterns of unknowns taken during the corresponding period. (Section 10.2, Method 100.2)

c) Spot Size

The diameter of the smallest beam spot at crossover must be measured regularly. Photograph the beam at crossover at 20,000 to 25,000X at s short exposure setting (to avoid spreading of the exposed spot on the film). Measure the diameter on the negative and divide by the magnification used. The resulting figure must be less than 250 nm. (Section 10.3, method 100.2)

d) EDXA System

The resolution and calibration of the energy dispersive X-ray analysis (EDXA) must be checked at least monthly and after service. The Al and Cu K" peaks should be centered at 1.48 KeV and 8.04 KeV respectively. The deviation from these energies should be no more than "10eV. The ability of the system to resolve the sodium K" line from the copper L line should be confirmed by obtaining a spectrum from a standard crocidolite sample on a copper grid. The k-factors relative to silicon should be calculated for Na, Mg, Al, Si, Ca, and Fe using NIST SRM 2063. The k-factor for Mg to Fe must be calculate; a value of 1.5 or less is required. (Section 10.4, Method 100.2)

D7.6 Detection Limits

A sensitivity of 200,000 fibers per liter (0.2 MFL) is required.

D7.7 Data Reduction

a) The concentration of asbestos in a given sample must be calculated. Refer to Section 5.10.6, “Computers and Electronic Data Related Requirements”, of this document.

c) Method Uncertainties - the laboratory must calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample. The laboratory shall have the ability to trace all sources of method uncertainties and their propagation to reported results. The ISO “Guide to the Expression of Uncertainty in Measurement” and/or the NIST Technical Note 1297 on “Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results” should be used in this regard.

D7.8 Quality of Standards and Reagents

a) The quality control program shall establish and maintain provisions for asbestos standards.

1) Reference standards that are used in an asbestos laboratory shall be obtained from the National Institute of Standards and Technology (NIST), EPA, or suppliers who participate in supplying NIST standards or NIST traceable asbestos. Any reference standards purchased outside the United States shall be traceable back to each country’s national standards laboratory. Commercial suppliers of reference standards should conform to ANSI N42.22 to assure the quality of their products.

2) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22-1995, Section 8, Certificates.

c) All reagents used shall be analytical reagent grade or better.

D7.9 Constant and Consistent Test Conditions

a) To prevent incorrect analysis results caused by the spread of contamination among samples, the laboratory shall establish and adhere to written procedures to minimize the possibilities of cross-contamination between samples.

b) Carbon-coating Filter Segments - Coating must be performed with a high-vacuum evaporation unit equipped with a rotating tilting stage. The carbon rods should be sharpened by a carbon rod sharpener to necks of about 4 mm long and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 10 cm from the surface of the microscope slide. (Section 11.18.1, Method 100.2)